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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:

REIYAO ZHU — CASE NO.: HT4000USNA

APPLICATION NO.: 10/803,384 — GROUP ART UNIT: 1771

FILED: MARCH 18, 2004 — EXAMINER: ANDREW T. PIZIALI

FOR: MODACRYLIC/COTTON/ARAMID FIBER BLENDS FOR ARC AND FLAME PROTECTION

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Reiyao Zhu, hereby declare:

THAT I hold a Doctorate of Philosophy (1991) in Textile Engineering from Leeds University, UK; a Master of Engineering and Bachelor of Science in Textile Science and Engineering from China Textile University of Shanghai, China. I have taught and conducted research in textile science and engineering at China Textile University, Bradford University (UK), and Texas Tech University.

That presently I am a Senior Research Engineer of the DuPont Personal Protection business of E. I. du Pont de Nemours and Company, and for the last 8 years have focused my research on high performance fibers, the development of fabrics for protective apparel, and protective apparel and garments.

That I am named an inventor on at least seven United States patents primarily dealing with high performance fibers, protective apparel fabrics, and protective apparel and garments.

That I am the inventor of U.S. Patent Application 10/803,384 filed March 18, 2004 (also referenced herein as my patent application).

That I have been advised that Smith, Jr. USP 4,865,906 has been applied in a rejection of claims in my patent application and I have been asked to determine if this publication has any applicability.

That I state Smith, Jr. USP 4,865,906 has no relevance to my patent application since for example this publication requires 25 to 85 weight % "oxidized polyacrylonitrile" while in contrast amended claim 1 of my patent application sets forth 45 to 60 weight % "modacrylic" (based on three components).

That I state that "oxidized polyacrylonitrile" fiber represents a carbonized material with a different chemical makeup and cannot be considered a "modacrylic" fiber.

That I provide the following explanation with an initial portion directed to a conventional definition of modacrylic fiber and the remaining portion of the explanation directed to carbon fiber formation including predominant use of polyacrylonitrile as a starting material:

Modacrylic fiber a manufactured fiber in which the fiberforming substance is any long chain synthetic polymer composed of less than 84% but at least 50% by weight of acrylonitrile units. (-CH2CH[CN]-)x. Modacrylic fibers are made from resins that are copolymers (combinations) of acrylonitrile and other materials, such as vinyl chloride, vinylidene chloride or vinyl bromide. Modacrylic fibers are either dry spun or wet spun. Modacrylic Fiber have characteristics of soft, resilien, easy to dye to bright shades, abrasion resistant, flame resistant, quick drying, resistant to acids and alkalies, shape retentive. It can be made to fabrics like fleece, knit-pile backing or nonwoven fabrics. It is suitable for making garment like deep-pile coats, trims and linings, simulated fur, wigs and hair pieces, children's sleepwear, career apparel. Fake Furs are often made out of this fiber.

A carbon fiber is a long, thin strand of material about 0.0002-0.0004 in (0.005-0.010 mm) in diameter and composed mostly of carbon atoms. The carbon atoms are bonded together in microscopic crystals that are more or less aligned parallel to the long axis of the fiber. The crystal alignment makes the fiber incredibly strong for its size. Several thousand carbon fibers are twisted together to form a yarn, which may be used by itself or woven into a fabric. The yarn or fabric is combined with epoxy and wound or molded into shape to form various composite materials. Carbon fiber-reinforced composite materials are used to make aircraft and spacecraft parts, racing car bodies, golf club shafts, bicycle frames, fishing rods, automobile springs, sailboat masts, and many other components where light weight and high strength are needed.

Raw Materials

The raw material used to make carbon fiber is called the precursor. About 90% of the carbon fibers produced are made from polyacrylonitrile. The remaining 10% are made from rayon or petroleum pitch. All of these materials are organic polymers, characterized by long strings of molecules bound together by carbon atoms. The exact composition of each precursor varies from one company to another and is generally considered a trade secret.

During the manufacturing process, a variety of gases and liquids are used. Some of these materials are designed to react with the fiber to achieve a specific effect. Other materials are designed not to react or to prevent certain reactions with the fiber. As with the precursors, the exact compositions of many of these process materials are considered trade secrets.

Manufacturing

Process

The process for making carbon fibers is part chemical and part mechanical. The precursor is drawn into long strands or fibers and then heated to a very high temperature without allowing it to come in contact with oxygen. Without oxygen, the fiber cannot burn. Instead, the high temperature causes the atoms in the fiber to vibrate violently until most of the non-carbon atoms are expelled. This process is called carbonization and leaves a fiber composed of long, tightly

Spinning

- 1 Acrylonitrile plastic powder is mixed with another plastic, like methyl acrylate or methyl methacrylate, and is reacted with a catalyst in a conventional suspension or solution polymerization process to form a polyacrylonitrile plastic.
- 2 The plastic is then spun into fibers using one of several different methods. In some methods, the plastic is mixed with certain chemicals and pumped through tiny jets into a chemical bath or quench chamber where the plastic coagulates and solidifies into fibers. This is similar to the process used to form polyacrylic textile fibers. In other methods, the plastic mixture is heated and pumped through tiny jets into a chamber where the solvents evaporate, leaving a solid fiber. The spinning step is important because the internal atomic structure of the fiber is formed during this process.
- 3 The fibers are then washed and stretched to the desired fiber diameter. The stretching helps align the molecules within the fiber and provides the basis for the formation of the tightly bonded carbon crystals after carbonization.

Stabilizing

- 4 Before the fibers are carbonized, they need to be chemically altered to convert their linear atomic bonding to a more thermally stable ladder bonding. This is accomplished by heating the fibers in air to about 390-590° F (200-300° C) for 30-120 minutes. This causes the fibers to pick up oxygen molecules from the air and rearrange their atomic bonding pattern. The stabilizing chemical reactions are complex and involve several steps, some of which occur simultaneously. They also generate their own heat, which must be controlled to avoid overheating the fibers. Commercially, the stabilization process uses a variety of equipment and techniques. In some processes, the fibers are drawn through a series of heated chambers. In others, the fibers pass over hot rollers and through beds of loose materials held in suspension by a flow of hot air. Some processes use heated air mixed with certain gases that chemically accelerate the stabilization.

Carbonizing

- 5 Once the fibers are stabilized, they are heated to a temperature of about 1,830-5,500° F (1,000-3,000° C) for several minutes in a furnace filled with a gas mixture that does not contain oxygen. The lack of oxygen prevents the fibers from burning in the very high temperatures. The gas pressure inside the furnace is kept higher than the outside air pressure and the points where the fibers enter and exit the furnace are sealed to keep oxygen from entering. As the fibers are heated, they begin to lose their non-carbon atoms, plus a few carbon atoms, in the form of various gases including water vapor, ammonia, carbon monoxide, carbon dioxide, hydrogen, nitrogen, and others. As the non-carbon atoms are expelled, the remaining carbon atoms form tightly bonded carbon crystals that are aligned more or less parallel to the long axis of the fiber. In some processes, two furnaces operating at two different temperatures are used to better control the rate de heating during carbonization.

Treating the surface

- 6 After carbonizing, the fibers have a surface that does not bond well with the epoxies and other materials used in composite materials. To give the fibers better bonding properties, their surface is slightly oxidized. The addition of oxygen atoms to the surface provides better chemical bonding properties and also etches and roughens the surface for better mechanical bonding properties. Oxidation can be achieved by immersing the fibers in various gases such as air, carbon dioxide, or ozone; or in various liquids such as sodium hypochlorite or nitric acid. The fibers can also be coated electrolytically by making the fibers the positive terminal in a bath filled with various electrically conductive materials. The surface treatment process must be carefully controlled to avoid forming tiny surface defects, such as pits, which could cause fiber failure.

Sizing

- 7 After the surface treatment, the fibers are coated to protect them from damage during winding or weaving. This process is called sizing. Coating materials are chosen to be compatible with the adhesive used to form

composite materials. Typical coating materials include epoxy, polyester, nylon, urethane, and others.

- 8 The coated fibers are wound onto cylinders called bobbins. The bobbins are loaded into a spinning machine and the fibers are twisted into yarns of various sizes.

That I state in summary Smith, Jr. USP 4,865,906 has no relevance to my patent application since oxidized polyacrylonitrile fiber is chemically different from modacrylic fiber.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Reiyao Zhu

REIYAO ZHU

9/22/06

DATED